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Contents

Articles

Chris E. Shuchart, Richard R. Willis, Andrew Wojcicki, Arnold L. Rheingold, Brian S. Haggerty

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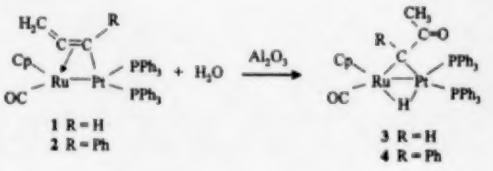
Conversion of the allenyl complexes $(PPh_3)_2Pt(\mu\text{-}\eta^1{:}\eta^2_{\alpha,\beta}\text{-}C(R){=}C{=}CH_2)Ru(CO)Cp$ to the hydrido-alkylidene complexes $(PPh_3)_2Pt(\mu\text{-}H)(\mu\text{-}\eta^1{:}\eta^1\text{-}C(R)C(O)CH_3)Ru(CO)Cp$ (R = H, Ph) by reaction with water under heterogeneous conditions

Reactions of the heterobimetallic PtRu μ-allenyl complexes **1** and **2** in CH_2Cl_2 or $CHCl_3$ solution with suspended deactivated alumina, silica or Florisil afford the corresponding μ-hydrido-μ-alkylidene products **3** and **4**. The conversion of **1** to **3** is very sensitive to reaction conditions: there is no reaction of **1** with $CHCl_3$–H_2O alone, and activated alumina, ether solvents and free radical inhibitors suppress this 'hydration'. Complex **3** was structurally characterized by single crystal X-ray diffraction analysis.

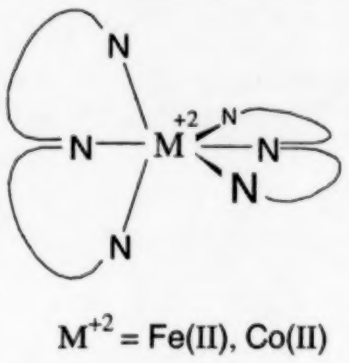


Tim Ayers, Sevena Scott, James Goins, Nicole Caylor, David Hathcock, Spencer J. Slattery, Donald L. Jameson

Inorg. Chim. Acta 307 (2000) 7

Redox and spin state control of Co(II) and Fe(II) *N*-heterocyclic complexes

A series of iron and cobalt compounds with the general formulas $[Fe(L)_2]^{2+}$ and $[Co(L)_2]^{2+}$, where L represents the tridentating ligands 2,2′:6′;2″-terpyridine, 6-(*N*-pyrazolyl)-2,2′-bipyridine, and 2,6-bis(*N*-pyrazolylpyridine, were prepared to investigate the extent of electronic regulation by varying the number of coordintaed pyridines relative to pyrazoles. The redox and spin state properties of both series are presented and compared.

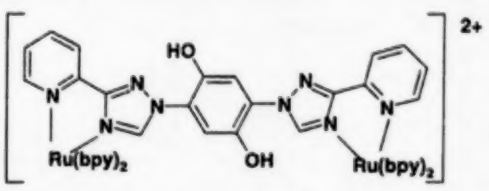


Frances M. Weldon, Johannes G. Vos

Inorg. Chim. Acta 307 (2000) 13

Synthesis and characterisation of ruthenium polypyridyl complexes containing a pendent hydroquinone grouping

The synthesis of the ligand 1,4-dihydroxy-2,5-bis(3-(pyridin-2-yl)-1,2,4-triazol-1-yl)benzene (**L1**) and its mono and dinuclear rutheniumbis(2,2′-bipyridyl) complexes is reported. The compounds obtained are characterised by NMR, UV–Vis and emission spectroscopy and have also been studied by electrochemical and spectroelectrochemical techniques. The data obtained suggest that **L1** is bound to the metal centre via the pyridine ring and the N4 atom of the triazole moiety. The compounds emit at room temperature and electrochemical studies show that the central hydroquinone unit is oxidised to a quinone moiety before the ruthenium centres.



Daniel Miguel, Víctor Riera, Mei Wang

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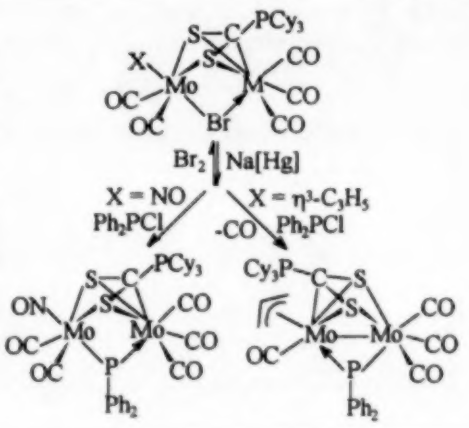
The influence of nitrosyl and allyl ligands on the formation of metal–metal bonds in bimetallic complexes containing S_2CPCy_3 bridges

Different products $[(ON)(CO)_2Mo(\mu\text{-}PPh_2)(\mu\text{-}S_2CPCy_3)Mo(CO)_3]$ and $[(\eta^3\text{-}C_3H_5)(CO)Mo(\mu\text{-}PPh_2)(\mu\text{-}S_2CPCy_3)Mo(CO)_3]$ were formed, respectively, by the reduction of the complexes $[X(OC)_2Mo(\mu\text{-}Br)(\mu\text{-}S_2CPCy_3)Mo(CO)_3]$ (X = NO, $\eta^3\text{-}C_3H_5$) and the subsequent reactions with Ph_2PCl. The electronic effects of the nitrosyl and allyl ligands have a significant influence on the formation of the metal–metal bond in the products containing both PPh_2 and S_2CPCy_3 bridges.



Rachel L. Fanshawe, Akbar Mobinikhaledi, Charles R. Clark, Allan G. Blackman

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Overcoming the chelate effect: hypodentate coordination of ethylenediamine, diethylenetriamine and tris(2-aminoethyl)amine in Co(III) complexes

The synthesis and characterisation of Co(III) complexes containing hypodentate polyamine ligands (en, dien, tren) is described, and crystal structures of *cis*-$[Co(en)_2(enH)imH]Br_4$ and $[Co(NH_3)_5enH]Cl_4$ are reported. These show that the monodentate en ligands adopt a *gauche* conformation in both structures.

Barbara Modec, Jurij V. Brenčič, Ljubo Golič, Gerald Giester

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Oxomolybdenum coordination compounds with pyridine. Syntheses and structures of $[Mo_4O_8(OCH_3)_2Cl_2L_4]\cdot 2L$ (L = Py, 4-MePy), $[Mo_4O_8(OCH_3)_4(4\text{-}MePy)_4]$, $[Mo_6O_{12}(OCH_3)_6Py_4]$ and $[Mo_{10}O_{26}Py_8]\cdot 7Py$

Reactions of $(PyH)_2[MoOCl_5]$ with methanol and pyridine or 4-methylpyridine under slightly different conditions have resulted in diamagnetic oxomolybdenum(V) and oxomolybdenum(V,VI) clusters whose d electrons are localized in Mo^V–Mo^V bonds i.e. $[Mo_4O_8(OCH_3)_2Cl_2Py_4]\cdot 2Py$, $[Mo_4O_8(OCH_3)_2Cl_2(4\text{-}MePy)_4]\cdot 2(4\text{-}MePy)$, $[Mo_4O_8(OCH_3)_4(4\text{-}MePy)_4]$, $[Mo_6O_{12}(OCH_3)_6Py_4]$ and $[Mo_{10}O_{26}Py_8]\cdot 7Py$.

Ela Solomon-Rapaport, Alexandra Masarwa, Haim Cohen, Dan Meyerstein

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Catalyzed autoxidation of azide by cobalt(II) (2,3,9,10-tetramethyl-1,4,8,11-tetraazacyclotetradeca-1,3,8,10-tetraene)$^{2+}$

In the presence of dioxygen Co(tim)$(H_2O)_2^{2+}$ reacts with azide in a complex process to produce $Co(tim)(N_3)_x^{(3-x)+}$ (x = 1 or 2) (λ_{max} at 340 nm) and dinitrogen. The reaction is clearly catalytic with up to several hundred turnovers. The dioxygen dependence of the process implies the involvement of an oxygen adduct, stabilized by an axial azide ligand.

$$[(N_3^-)Co^{II}(tim)(H_2O)]^+ + O_2 \leftrightarrows [(N_3^-)\text{-}Co^{III}(tim)\text{-}O_2^-]^+$$

$$[(N_3^-)\text{-}Co^{III}(tim)\text{-}O_2^-]^+ + N_3^- + 2\,H^+ \rightarrow Co^{II}(tim)^{2+} + 3\,N_2 + H_2O_2$$

Leeni Hirsivaara, Lucia Guerricabeitia, Matti Haukka, Pekka Suomalainen, Riitta H. Laitinen, Tapani A. Pakkanen, Jouni Pursiainen

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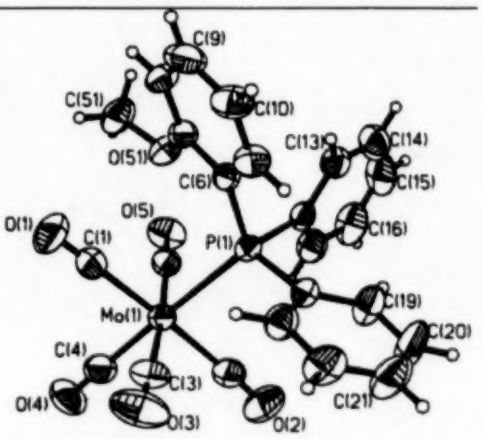
$M(CO)_6$ (M = Cr, Mo, W) derivatives of (*o*-anisyl)-diphenylphosphine, bis(*o*-anisyl)phenylphosphine tris(*o*-anisyl)phosphine and (*p*-anisyl)bis(*o*-anisyl)phosphine

$Cr(CO)_6$, $Mo(CO)_6$ and $W(CO)_6$ derivatives of *ortho*-methoxy substituted aromatic tertiary phosphine ligands are prepared and characterized by X-ray crystallography, ^1H, $^{13}C\{^1H\}$ and $^{31}P\{^1H\}$ NMR spectroscopy, IR spectroscopy and elemental analysis. The steric properties of the ligands are studied by molecular modelling methods.



Zheng Wang, Yousheng Guan, Phillip E. Fanwick, Joseph G. Stowell, Donald E. Bergstrom, Mark A. Green

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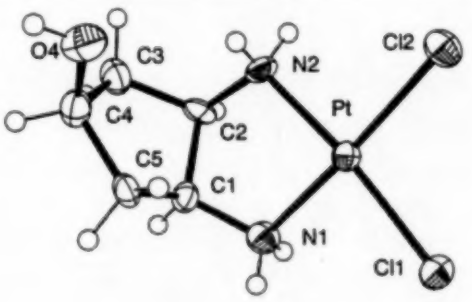
cis-3,4-Diaminocyclopentanol complexes of platinum(II)

cis-$[PtCl_2$(*cis*-1,2-diaminocyclopentane)] (**A**), *cis*-$[PtCl_2$(*cis*,*syn*-3,4-diaminocyclopentanol)] (**B**), *cis*-$[PtCl_2$(*cis*,*anti*-3,4-diaminocyclopentanol)] (**C**), and bis(*cis*,*anti*-3,4-diaminocyclopentanol)platinum(II) tetrachloroplatinate (**D**), have been synthesized and structurally characterized by X-ray crystallography.



Scott W. Schweiger, Emily E. Freeman, Janet R. Clark, Mark C. Potyen, Phillip E. Fanwick, Ian P. Rothwell

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Studies of six-coordinate adducts all-*trans*-[M-$(OAr)_2(X)_2(L)_2$] (M = Nb, Ta; X = Cl, Br; L = tertiary phosphine, pyridine)

An extensive series of d^1-derivatives of niobium and tantalum $[M(OAr)_2(X)_2(L)_2]$ (M = Nb, Ta; X = Cl, Br; L = tertiary phosphine, pyridine) have been isolated by reduction of the corresponding adducts $[M(OAr)_2(X)_3(L)]$ in the presence of excess L. Crystallographic studies show an all-*trans*, octahedral arrangement of ligands about the metal center. The effect of the halide, donor ligand and aryloxide substituents upon the structural parameters is discussed.

Arvydas Tamulis, Rumiya R. Abdreimova, Jelena Tamuliene, Maurizio Peruzzini, Mindaugas L. Balevicius

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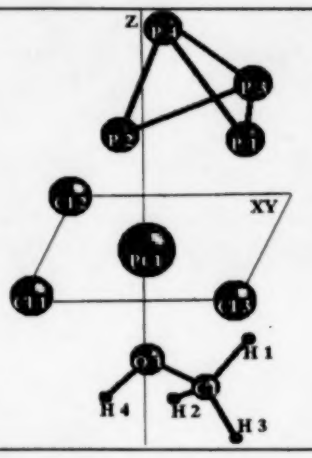
Platinum-mediated oxidative P–O coupling of white phosphorus and alcohols: an ab initio study

Better conditions for the nucleophilic attack of the P_4 molecule by the alkoxide ion, that is originated from the coordinated CH_3OH molecule, are found in the cationic Pt(IV) η^2-P_4 complexes. The halide ligands, which complete the coordination sphere around Pt, promote the reaction via nucleophilic assistance to the alcohol deprotonation step.



Ivan M. Lorković, Xianhui Bu, Peter C. Ford

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Synthesis and characterization of the dinuclear iron–sulfur cluster {bis[(1,1′-dithiolatoferrocene-μ-sulfido-ferrate(III)]}$^{2-}$

The novel iron–sulfur cluster dimer {bis-[(1,1′-dithiolatoferrocene-μ-sulfido-ferrate-(III)]}$^{2-}$ is prepared as the tetrapropylammonium salt in a one-pot synthesis from 1,1′-trithiaferrocenophane, $FeCl_3$ and $^tBuS^-$. The X-ray crystallography, electronic spectroscopy, electrochemistry and magnetic susceptibility of this complex are reported and compared and contrasted to previously prepared iron–sulfur dimer complexes with organic thiolate ligands.

Dave R. van Staveren, Jaap G. Haasnoot, Anna Maire Manotti Lanfredi, Stephan Menzer, Peter J. Nieuwenhuizen, Anthony L. Spek, Franco Ugozzoli, Jan Reedijk

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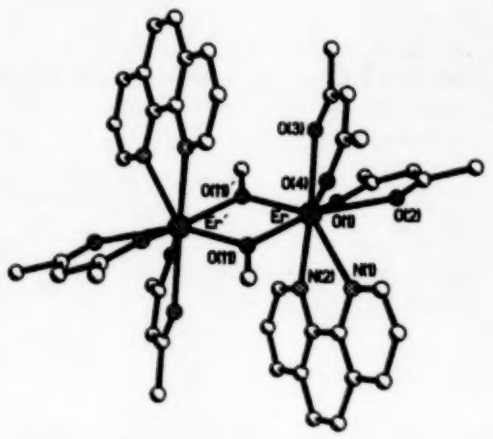
Synthesis and X-ray crystal structures of two novel dinuclear methoxo-bridged hexafluoropentanedionato Er(III) complexes with 1,10-phenanthroline and 2,2′-bipyridine

The first two examples of structurally characterised eight-coordinated dinuclear methoxo-bridged lanthanide complexes are reported. The two compounds $[Er(hfpd)_2(\mu\text{-}OCH_3)(bpy)]_2$ and $[Er(hfpd)_2(\mu\text{-}OCH_3)(phen)]_2$ were obtained from reactions of $Er(CF_3SO_3)_3$ with hexafluoropentane-2,4-dione, $CsOH\cdot H_2O$ and either 2,2′-bipyridine or 1,10-phenanthroline in MeOH and were characterised by X-ray crystallography, IR and far-IR spectroscopy.



Xiaoyuan Chen, Frank J. Femia, John W. Babich, Jon Zubieta

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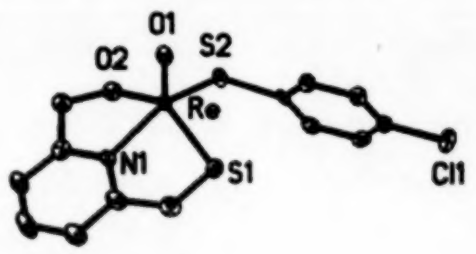
Synthesis and characterization of oxorhenium(V)–'3 + 1' mixed thiolate [SNS]/[S] and [ONS]/[S] complexes. Crystal and molecular structures of $[ReO(\eta^3\text{-}SCH_2C_5H_3NCH_2S)(\eta^1\text{-}C_6H_4Br\text{-}4\text{-}S)]$, $[ReO(\eta^3\text{-}SCH_2C_5H_3NCH_2O)(\eta^1\text{-}C_6H_4X\text{-}4\text{-}S)]$ (X = Cl, OMe), $[ReO(\eta^3\text{-}SCH_2C_5H_3NCH_2O)(\eta^1\text{-}C_6H_4OCH_3\text{-}4\text{-}CH_2S)]$ and $[ReO(\eta^3\text{-}SCH_2C_5H_3\text{-}NCH_2S)(\eta^1\text{-}C_5H_4NH\text{-}2\text{-}S)][Cl]$

The reaction of $[ReOCl_3(PPh_3)_2]$ with the tridentate ligands 2,6-dithiomethylpyridine (**4**) or 6-thiomethyl-2-pyridinemethanol (**5**) and the appropriate monothiols in the presence of Et_3N leads to the isolation of a series of integrated '3 + 1' oxorhenium(V) complexes. Similarly, reaction of $[ReOCl_3(PPh_3)_2]$ with 2-mercaptopyridine and **4** afforded a cationic oxorhenium(V) complex, $[ReO(\eta^3\text{-}SCH_2C_5H_3NCH_2S)(\eta^1\text{-}C_5H_4NH\text{-}2\text{-}S)][Cl]$ (**22**).



Fabio Marchetti, Claudio Pettinari, Riccardo Pettinari, Donatella Arriva, Sergei Troyanov, Andrei Drozdov

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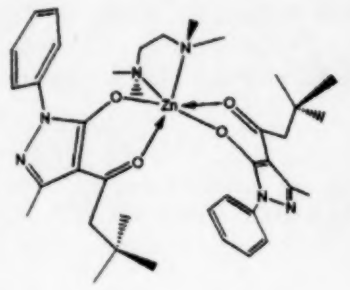
On the interaction of acylpyrazolonates with zinc(II) acceptors: the role of ancillary ligands

Synthesis and characterisation of several bis(acylpyrazolonate)zinc(II) derivatives with ancillary N-, O- and S-donor ligands. They are five- or six-coordinate zinc complexes which, in the presence of acidic H atoms in ancillary ligands, show the solid state supramolecular architecture influenced by extended intermolecular hydrogen bonding involving the acceptor N atoms in acyl pyrazolonato ligands.

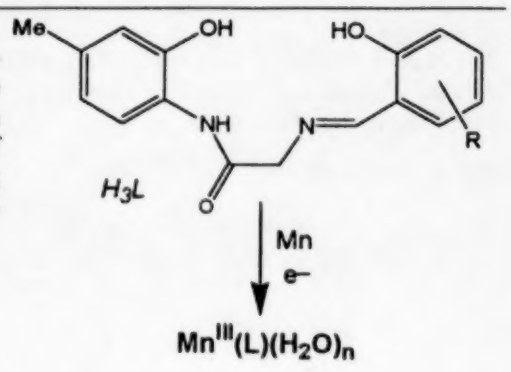


M.R. Bermejo, A.M. González, M. Fondo, M. Maneiro, M. Rey, M. Vázquez, O.L. Hoyos, J.C. García-Monteagudo

Inorg. Chim. Acta 307 (2000) 106

Electrochemical synthesis of neutral Mn(III) complexes with asymmetrical N_2O_2 Schiff base ligands

A new series of potentially tetradentate trianionic Schiff bases has been prepared. The electrochemical oxidation of manganese in the presence of an acetonitrile solution of these ligands presents a simple and efficient route to obtain neutral manganese(III) complexes with a high yield and purity.



Petra Annen, Sandra Schildberg, William S. Sheldrick

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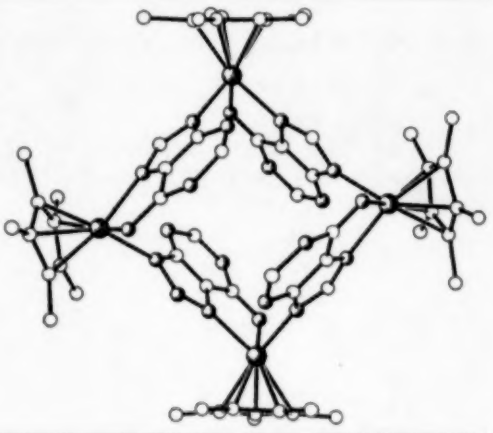
(η^5-Pentamethylcyclopentadienyl)iridium(III) complexes of purine nucleobases and nucleotides: a comparison with (η^6-arene)ruthenium(II) and (η^5-pentamethylcyclopentadienyl)rhodium(III) species

$[(\eta^5\text{-}C_5Me_5)Ir(H_2O)_3](CF_3SO_3)_2$ reacts with adenine (AH) and guanine (GH) to afford cyclic tetranuclear cations $[\{(\eta^5\text{-}C_5Me_5)Ir(A)\}_4]^{4+}$ **(1)** and $[\{(\eta^5\text{-}C_5Me_5)Ir(G)(H_2O)\}_4]^{4+}$ **(3)** with $\mu\text{-}N^7,N^9$ bridging. Treatment of this organometallic tris-aqua complex with 5′-ATP^{4-} and 5′-ITP^{4-} in the pH range 4–7 yields diastereomeric $\mu\text{-}N^1,N^7$ bridged cyclic trimers in contrast to the (η^5-C_5Me_5)Rh(III) and (η^6-C_6H_6)Ru(II) fragments for which phosphate-coordinated species predominate.



Notes

Antonio Romerosa, Carlos López-Magaña, Mustapha Saoud, Sonia Mañas, Enrique Colacio, José Suárez-Varela

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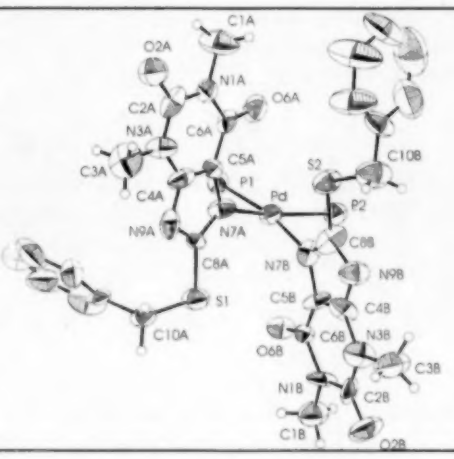
Palladium phosphine complexes from 8-(thio)-theophylline, 8-(methylthio)-theophylline and 8-(benzylthio)-theophylline

The synthesis and the characterization of 8-(benzylthio)-theophylline (8-BzTTH), a new organic ligand based on the molecular skeleton of 8-thio-purine, and a new procedure to obtain 8-(methylthio)-theophylline (8-MTTH) are presented together with a study describing their reactivity and the reactivity of 8-(thio)-theophylline (8-TTH_2) with [*cis*-$PdCl_2(PPh_3)_2$]. The reactions of 8-TTH_2, 8-MTTH and 8-BzTTH ligands with [*cis*-$PdCl_2(PPh_3)_2$] yield different Pd complexes depending upon the particular ligand and molar ratio.



Guan Hong Chu, Jung Bok Park, Minserk Cheong

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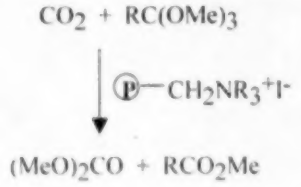
Synthesis of dimethyl carbonate from carbon dioxide over polymer-supported iodide catalysts

Polymer-supported iodide catalysts are effective heterogeneous catalysts for the synthesis of dimethyl carbonate via carbon dioxide activation in the presence of trimethyl orthoesters.



Scott E. Watkins, Donald C. Craig, Stephen B. Colbran

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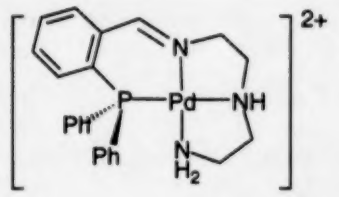
A palladium(II) complex of a new iminophosphine ligand derived from diethylenetriamine and 2-(diphenylphosphino)benzaldehyde

The square planar palladium(II) cation shown formed in high yield when [Pd(oP-$CHO)_2Cl_2$] [oPCHO = 2-(diphenylphosphino)benzaldehyde] and [Zn(dien)Cl_2] were reacted together in an unsuccessful attempt to template the formation of a bimetallic iminophosphine-bridged complex. The crystal structure of the PF_6^- salt of the cation is reported.



Charles S. Weinert, Charlotte L. Stern, Duward F. Shriver

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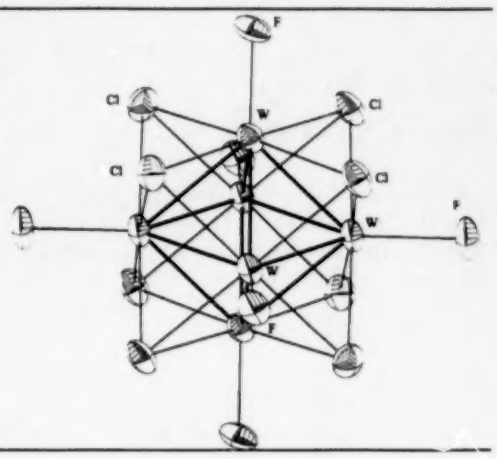
Preparation of $[Bu_4N]_2[W_6Cl_8F_6]$ and characterization of the clusters $[Bu_4N]_2[W_6Cl_8X_6]$ (X = F, Cl, Br, I, NCO, NCS, NCSe, or OSO_2CF_3) by ^{183}W NMR spectroscopy

The cluster $[Bu_4N]_2[W_6Cl_8F_6]$ (**1**) has been prepared from $[Bu_4N]_2[W_6Cl_6(OSO_2CF_3)_6]$ (**2**) in modest yield. A crystal structure has been determined for **1**. Additionally, the clusters $[Bu_4N]_2[W_6Cl_8X_6]$ (X = F, Cl, Br, I, NCO, NCS, NCSe, or OSO_2CF_3) have been characterized by ^{183}W NMR spectroscopy.

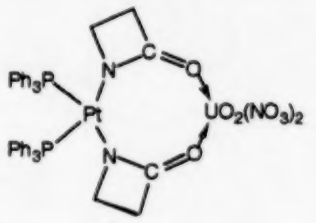


William Henderson, Allen G. Oliver, Clifton E.F. Rickard

Inorg. Chim. Acta 307 (2000) 144

Synthesis and structure of the amidate-bridged platinum–uranium bimetallic complex $[Pt\{\overline{NC(O)CH_2CH_2}\}_2(PPh_3)_2]\cdot UO_2(NO_3)_2$

The platinum amidate complex $[Pt\{\overline{NC(O)CH_2CH_2}\}_2(PPh_3)_2]$ acts as a metalloligand towards uranyl nitrate, giving $[Pt\{\overline{NC(O)CH_2CH_2}\}_2(PPh_3)_2]\cdot UO_2(NO_3)_2$, which has been structurally characterised.



Xiaoyuan Chen, Frank J. Femia, John W. Babich, Jon Zubieta

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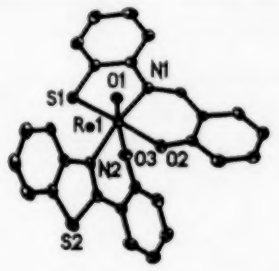
Schiff base chemistry of the $\{ReO\}^{3+}$ core: structural characterization of the unusual '3 + 2' complex $[ReO(\eta^3\text{-}OC_6H_4\text{–}CH{=}NC_6H_4\text{-}2\text{-}S)(\eta^2\text{-}OC_6H_4\overline{C{=}NC_6H_4\text{-}2\text{-}S})]$

$[ReOCl_3(PPh_3)_2]$ reacts with salicylaldehyde-2-mercaptoanil (**1**) to give $[ReO(OCH_3)(PPh_3)(OC_6H_4CH{=}NC_6H_4S)]$ (**3**), which reacts with a second equivalent of **1** to give $[ReO(\eta^3\text{-}OC_6H_4CH{=}NC_6H4_S)(\eta^2\text{-}OC_6H_4\overline{C{=}NC_6H_4S})]$ (**4**).



Xiaoyuan Chen, Frank J. Femia, John W. Babich, Jon A. Zubieta

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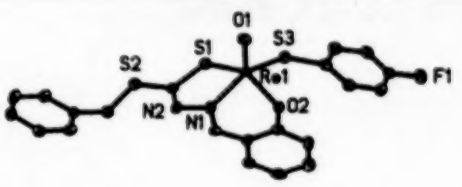
Exploring oxorhenium '3 + 1' mixed-ligand complexes carrying the *S*-benzyl-3-[(2-hydroxyphenyl)methylene]dithiocarbazate [ONS]/monothiol [S] donor set: synthesis and characterization

The reaction of $ReOCl_3(PPh_3)_2$ with the potentially tridentate ligand *S*-benzyl-3-[(2-hydroxyphenyl)methylene]dithiocarbazate [ONS] donor and *para*-substituted benzenethiols [C_6H_4X-4-SH] (where X = H, F, Cl, Br and OCH_3) afforded a series of '3 + 1' oxorhenium(V) complexes. The molecular structure of $[ReO\{\eta^3\text{-}OC_6H_4\text{-}2\text{-}CH{=}N{-}N{=}C(SCH_2Ph)\text{-}S\}(\eta^1\text{-}C_6H_4F\text{-}4\text{-}S)]$ (**4**) was determined by single-crystal X-ray analysis. Complex **4** consists of a central oxorhenium(V) core coordinated to the phenolic oxygen, azomethine nitrogen and thiol sulfur from the thiosemicarbazone Schiff base ligand and one sulfur donor from monothiol ligand in a distorted square pyramidal environment.



Frank J. Femia, Xiaoyuan Chen, Kevin P. Maresca, John W. Babich, Jon Zubieta

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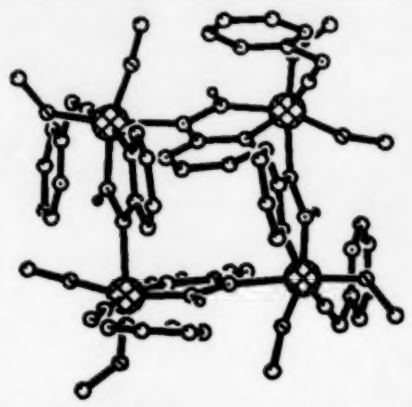
Synthesis and crystal and molecular structure of a tetranuclear cluster based on the rhenium(III)-bisorganohydrazino core: $[Re(HNNC_4H_3N_2)(NNC_4H_3N_2)(OCH_3)_2]_4$

Slow crystallization of $[Re(HNNC_4H_3N_2)(NNC_4H_3N_2H)Cl_3]$ (**1**) in methanol yields the tetranuclear complex $[Re(HNNC_4H_3N_2)(NNC_4H_3N_2)(OCH_3)_2]_4$ (**2**).



Kuninobu Kasuga, Kenji Nishie, Makoto Handa, Tamotsu Sugimori

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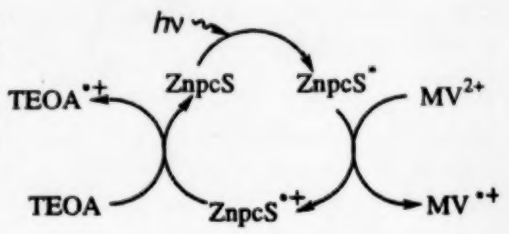
Determination of quenching process for the photoreduction of methyl viologen catalyzed by (sulfonatophthalocyaninato)zincate(II) in the presence of triethanolamine

Comparing an action spectrum for the photoreduction of MV^{2+} in a three-component system of TEOA, the title complex (ZnpcS), and MV^{2+} with absorption and excitation spectra of the complex, a quenching process for the system was determined.



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